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Research Article

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Isolation of Hex-2-ulofuranosyl hexopyranoside and GC-MS profile of different extracts from *Capparis decidua* (Forssk.)

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ABSTRACT

In this phytochemical studies Hex-2-ulofuranosyl hexopyranoside (sucrose) was been isolated from the aerial parts of Capparis decidua. The compound has been identified by different spectroscopic techniques including X-ray crystallography. The chloroform and ethylacetate extracts of the plant were further subjected to GC-MS which result in the identification of interesting compounds.

Key words: Hex-2-ulofuranosyl hexopyranoside, GC-MS profile, *Capparis decidua*

INTRODUCTION

Capparis decidua Family (Capparaceae) is widely distributed in typical deserts and semi- deserts areas in northern and central Sudan, especially on sandy soils and in low rainfall savanna on clays [1]. It is also found in Blue Nile, Upper Nile, western and eastern Sudan besides northern areas of the country [2]. In Sudan *C.decidua* is used traditionally as anti helmintic, analgesic, aphrodisiac, carminative, diaphoretic, emmenagogue and laxative. The bark extract is used for asthma and cough management. The paste of young leaves and branches are applied as protective coat on boils and swelling for the absorption of serious secretions [3], and for their anti-inflammatory, astringent effects, they are used also as stomachic, laxative, antidote, and used for skin diseases. The poultices of the twigs are used against head-ache [5]. Decoction preparation from the roots is used to relieve fever and is also used for jaundice. As fumigation, roots are used to treat fever and rheumatism [6]. The aerial part is used for rheumatism, gout; externally the infusion is used for boils, eruption and ulcers, while internally as antidote to poisons [7].

EXPERIMENTAL SECTION

2-1. Plant material

Capparis decidua (aerial parts) was collected from Shambat, Khartoum north-Sudan. The plant was authenticated at the Medicinal and Aromatic Plant Research Institute (MAPRI), Sudan and voucher specimens deposited in the Herbarium.

2-2. Extraction and isolation procedure

Collected plant was dried under shade and then powdered. The powdered plant material (200 gram) was extracted by cold maceration method with sufficient quantity of 80% methanol for 48 hrs. The process of extraction was repeated twice for the completion of the extraction. The extract was filtered using Whatman filter paper and the filtrates were concentrated under reduced pressure which afforded 39 g of a concentrated extract. A 30 g of this extract was fractionated on a normal phase silica gel column eluted with petroleum ether-chloroform and chloroform–ethyl acetate mixtures of increasing polarity to give the sub-fractions (1, 2 and 3). Compound MW-1 was obtained as colourless crystaline solid from the fraction of 50% methanol in ethylacetate during elution of coloumn packed with methanol extract of *Capparis decidua*. The crystals were collected and washed several times with methanol.

2-3. X-Ray crystallography

Conducted at X-Ray crystallography apparatus (diffractorometer with Molybdenum source). Bruker SMART APEX-Germany

2-4. Gas chromatography- Mass spectrophotometry (GC-MS) of different sub-fractions

GC-MS were carried out on the Schimadzu GCMS-QP1020 spectrometer operating at 45 to 500 MHz. 1 ml of compound being tested was dissolved in methanol. The solution was filtered through micro filter. Then 1 microliter of the solution was injected using Hamilton microliter syringe. The runs on the GC was done according to the following method:

Ionization technique: Electron Impact (EI). Carrier gas: Helium. Total flow rate: 50 ml/minute. Column flow rate: 1.6 ml/minute. Column: Capillary Column-DB5 (30m×0.25mm). Injection volume: 1 µl. Injection temperature: 240 ^{0}C Temperature program: Programmed at 50-3000C.

2-5. Nuclear magnetic resonance (NMR)

^{1}H-NMR- and ^{13}C-NMR spectra were carried out on the Bruker AM 500 and 700 spectrometer operating at 500 and 700 MHz (^{1}HNMR) in spectroscopic grade solvents D_2O, MeOD and $CDCl_3$. The chemical shifts values are expressed in δ (ppm) units using (TMS) as an internal standard and the coupling constants (*J*) are expressed in Hertz (Hz). Standard pulse sequences were used for generating COSY, HMQC and HMBC spectra (2D experiments).

5 mg of the compound being tested was dissolved in 0.6 ml of suitable solvent used and the experiments were sent to NMR instrument at temperature of 296.1 K.

RESULTS AND DISCUSSION

3-1. Structure Elucidation of Compound MW-1

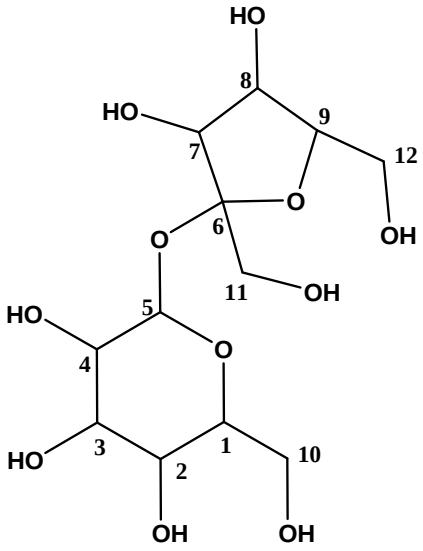
Compound **-1** was identified by spectroscopic technique including (1D and 2D NMR) and confirmed by X-ray crystallography.

Compound MW-1, was obtained as colorless crystalline solid from the fraction of 50% methanol in ethyl acetate during column elution. The ^{1}H NMR spectrum of MW-1 showen one proton doublet at δ 4.16 (J = 8.5Hz), another proton showen a double doublets at δ 4.00 (J = 4, 9 Hz) ascribed to oxygenated methane is assigned to H_7 and H_8 respectively. One proton shown a broad singlet at δ 5.36 is assigned to anamoric proton (H_5). While a third proton shown a double doublets at δ 3.43 (J = 9. 9 Hz) ascribed to oxygenated methine proton is assigned to H_4. Three set of protons (2 each) showen a broad multiplet at δ 3.76, 3.68 and a doublet at δ 3.51 (J = 9.5 Hz) were attributed to oxygenated methylene H_{12}, H_{10} and H_{11} protons respectively. Four set of protons (1 each) showen multiplets at δ 3.72- 4.17 were accounted to the remaining protons.^{13}C-NMR data showen a clear signals attributed to quaternary carbons at δ 103.68 is assigned to C_5. One anamoric carbon at δ 92.19 is assigned to C_6. Three oxygenated methylene carbons at δ 60.14, 61.38 and 62.38 are assigned to C_{10}, C_{12}, and C_{11} respectively. Six oxygenated methine carbons at δ 72.42, 69.24, 72.58, 71.08, 76.47, 74.02 and δ 81.37 are assigned to C_1, C_2, C_3, C_4, C_7, C_8 and C_9 respectively. The DEPT 90 spectrum of MW-1 showed that the signals at δ 69.2, 71.08, 72.4, 72.58, 74.02, 76.47 and 92.19 were related to the oxygenated methine protons.

The existence of ^{1}H NMR signals in the deshielded region at δ 5.36 and ^{13}C NMR signals at δ 92.19 and 103.68 supported the linkages of the two sugar units. The ^{1}H-^{1}H Cosy spectrum of MW-1 showed correlations between H_4 with H_3 and H_7 with $H_{8.}$

The HMBC spectrum of MW-1 exhibited interactions of H_9 (δ 3.62) with C_7 (δ76.47) by three bonds correlation, C_8 (δ 74.02) by tow bonds correlation and C_6 (δ 103.68) by four bonds correlation. In HSQC spectrum of MW-1, C_2 at δ 69.24 interacted with H_2 at δ 3.70; C_4 at δ 71.08 with H_4 at δ 3.43; C_1 at δ 72.42 with H_1 at δ 3.83; C_3 at δ 72.58 with H_3 at δ 3.72; C_8 at δ 74.02 with H_8 at δ 4.00; C_7 at δ 76.47 with H_7at δ 4.16; and C_9 at δ 81.37 with H_9 at δ 3.62. The X-ray crystallography of compound MW-1 clearly shows the structure of the compound as well as the bond orientation (fig-18). On the bases of these evidences the structure of MW-1 has been established as Hex-2-ulofuranosyl hexopyranoside [Sucrose] ($C_{12}H_{22}O_{11}$).

X-ray crystallography also indicates clearly the actual structure of the compound confirming our prediction.



Hex-2-ulofuranosyl hexopyranoside
[Sucrose]

Table-1: ^{1}H and ^{13}C NMR Spectral Data of Compound-1 (500 MHz, D_2O)

Position	δ ^{1}H (Multiplicity, *J* in Hz)	δ ^{13}C
1	3.83 m	72.42
2	3.70 m	69.24
3	3.72 m	72.58
4	3.43 dd (J=9, 9)	71.08
5	5.36 br	92.19
6	-	103.68
7	4.16 d (J=8.5)	76.47
8	4.00 dd (J=4, 9)	74.02
9	3.62 m	81.37
10	3.68 m	60.14
11	3.51 d (J=9.5)	62.38
12	3.76 m	61.38

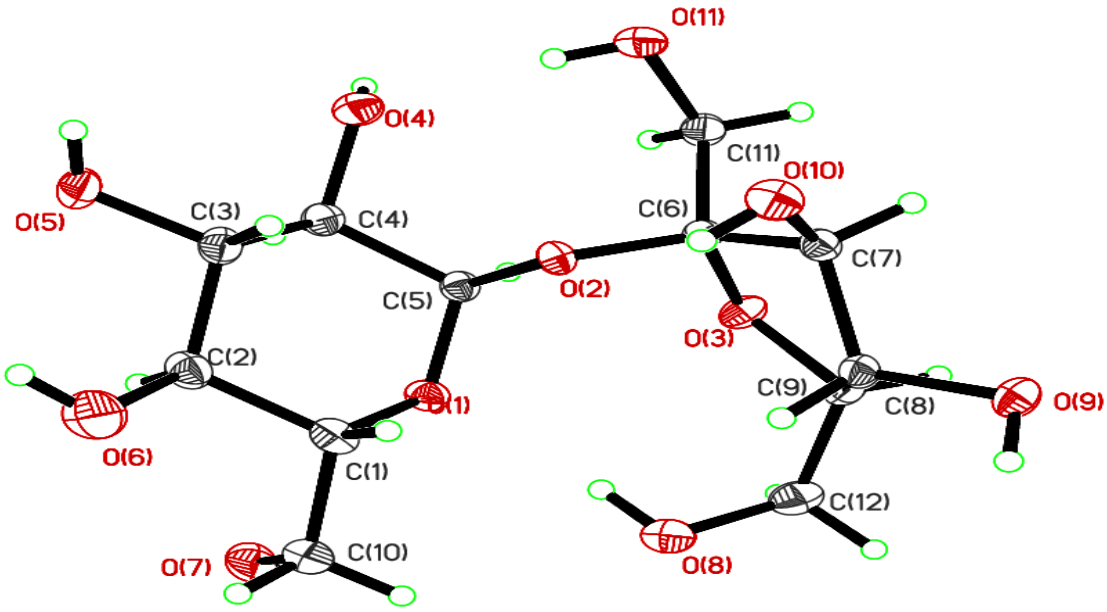


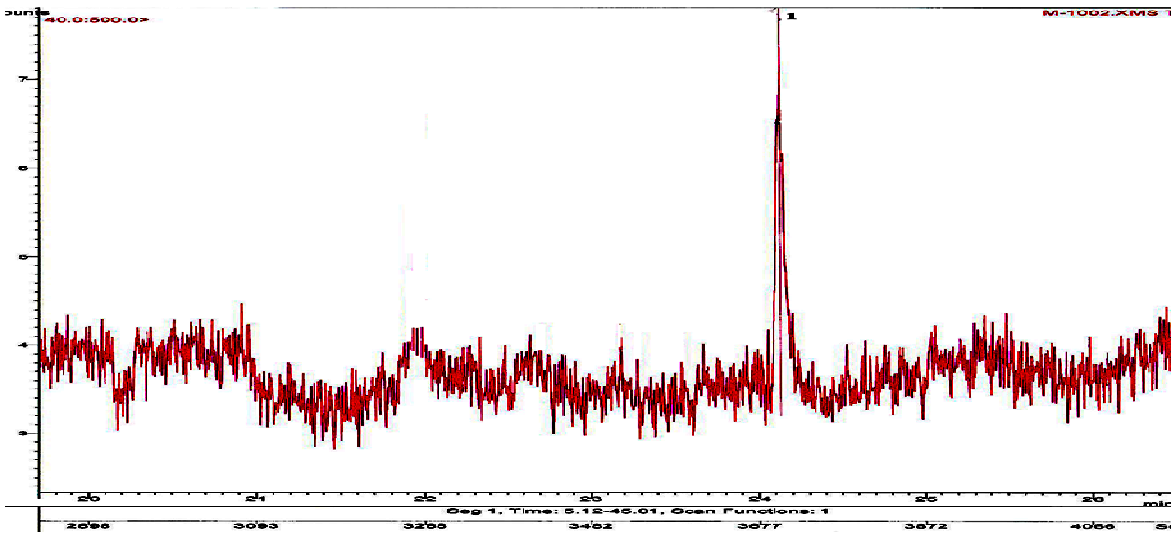
Fig-1: X-ray crystallography of compound -1

3-2. GC-MS analysis of *Capparis decidua* methanolic sub-fractions (1,2 and 3)

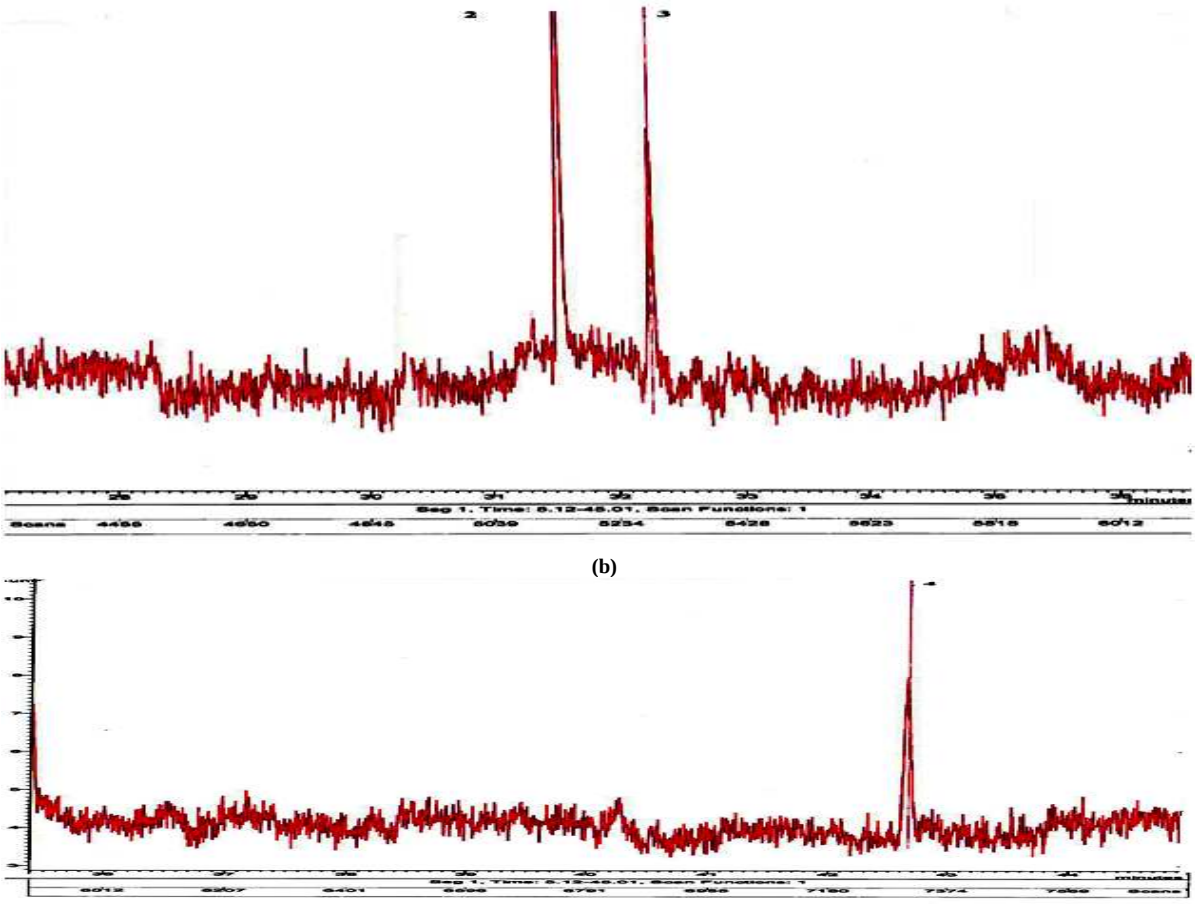
The GC chromatogram of Sub-fraction (1), exhibited four peaks at retention times (Rt) 24.129, 31.549, 32.268 and 42.664 min indicating presence of four compounds, while MS spectrum displayed $[M]^+$ at 177, 185, 150 and 275 m/z respectively, corresponding to different molecular ions. Suggestion of the separated components was accomplished using computer search by matching spectra with reference spectra in the computer library. The suggested compounds are shown in Table-2.

Table-2: GC-MS Analysis of SF-1

Compound number	Retention time in min. (R_t)	Compound name	Chemical formula	Base peak
1	24.129	Diethyl Phthalate	$C_{12}H_{14}O_4$	149
2	31.549	Tridecanoic acid	$C_{14}H_{28}O_2$	74
3	32.268	Dibutyl Phthalate	$C_{16}H_{22}O_4$	149
4	42.664	1,2-benzenedicarboxylic acid,diisooctyl ester	$C_{24}H_{38}O_4$	149



(a)



(b)

(c)

Fig-2: a, b and c; GC chromatogram of SF-1

The GC chromatogram of SF-2 exhibited four peaks at retention times (R_t) 28.734, 32.879, 36.562 and 39.945 min indicating presence of four compounds, while the MS spectrum displayed $[M]^+$ at 125, 125, 125 and 392 m/z respectively, corresponding to different molecular ions. The suggested compounds are shown in Table-3.

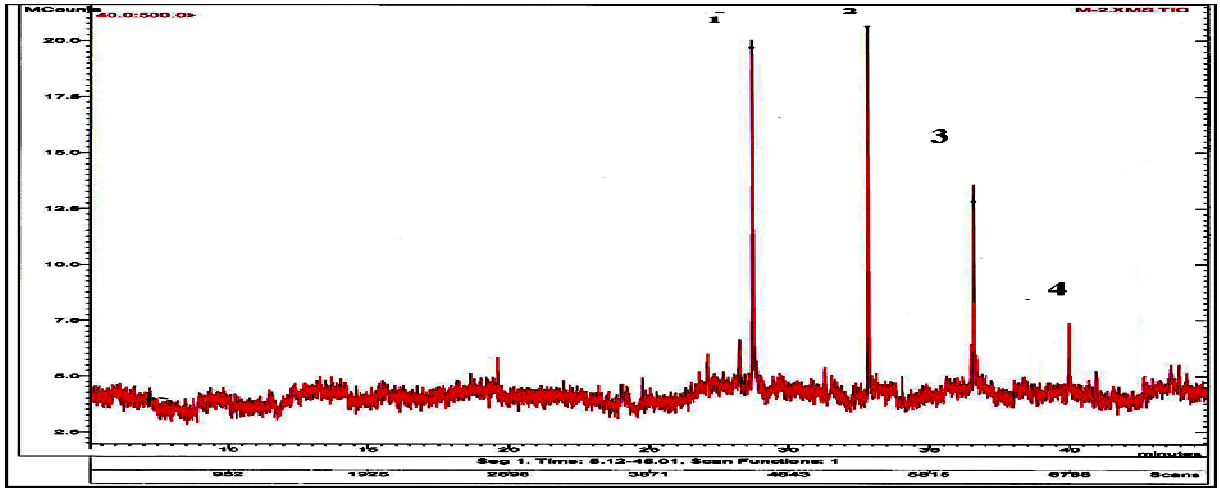


Fig-3: GC chromatogram of SF-2

Table-3: GC-MS Analysis of SF-2

Compound number	Retention time in min. (R_t)	Compound name	Chemical formula	Base peak
1	28.734	E-15-Heptadecenal	$C_{17}H_{32}O$	43.0
2	32.879	E-14-Hexadecenal	$C_{16}H_{30}O$	43,0
3	36.562	1-Docosene	$C_{22}H_{44}$	55.0
4	39.945	Cyclooctacosane	$C_{28}H_{56}$	57.0

The GC chromatogram of SF-3, exhibited three peaks at retention times (R_t) 29.923, 28.713, and 32.839 min indicating presence of three compounds, MS spectrum displayed $[M]^+$ at 206, 125 and 125 m/z respectively, corresponding to different molecular ions. The suggested compounds are shown in Table-4.

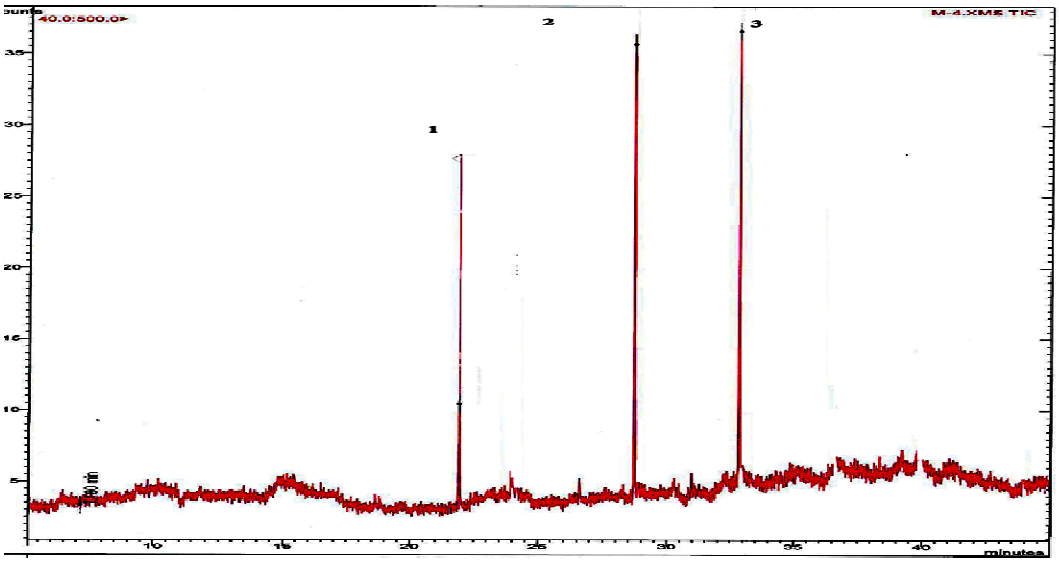


Fig-4: GC chromatogram of SF-3

Table-4: GC-MS Analysis of SF-3

Compound number	Retention time in min. (R_t)	Compound name	Chemical formula	Base peak
1	21.923	2,5-di-tert-butyl phenol	$C_{14}H_{22}O$	191.0
2	28.714	5-Ecosene	$C_{17}H_{34}$	55
3	32.839	E-16-Heptadecenal	$C_{17}H_{32}O$	43

CONCLUSION

Hex-2-ulofuranosyl hexopyranoside (sucrose) was isolated from *Capparis decidua* (Forssk.) for the first time, where an advanced spectrospical techniques were used for its identification. GC-MS technique one of the most powerful methods used for identification of active medicinal plants constituents, in addition it may help in assessing of quality against adulterant and act as a biochemical marker for those medicinally important plants in the pharmaceutical industry.

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